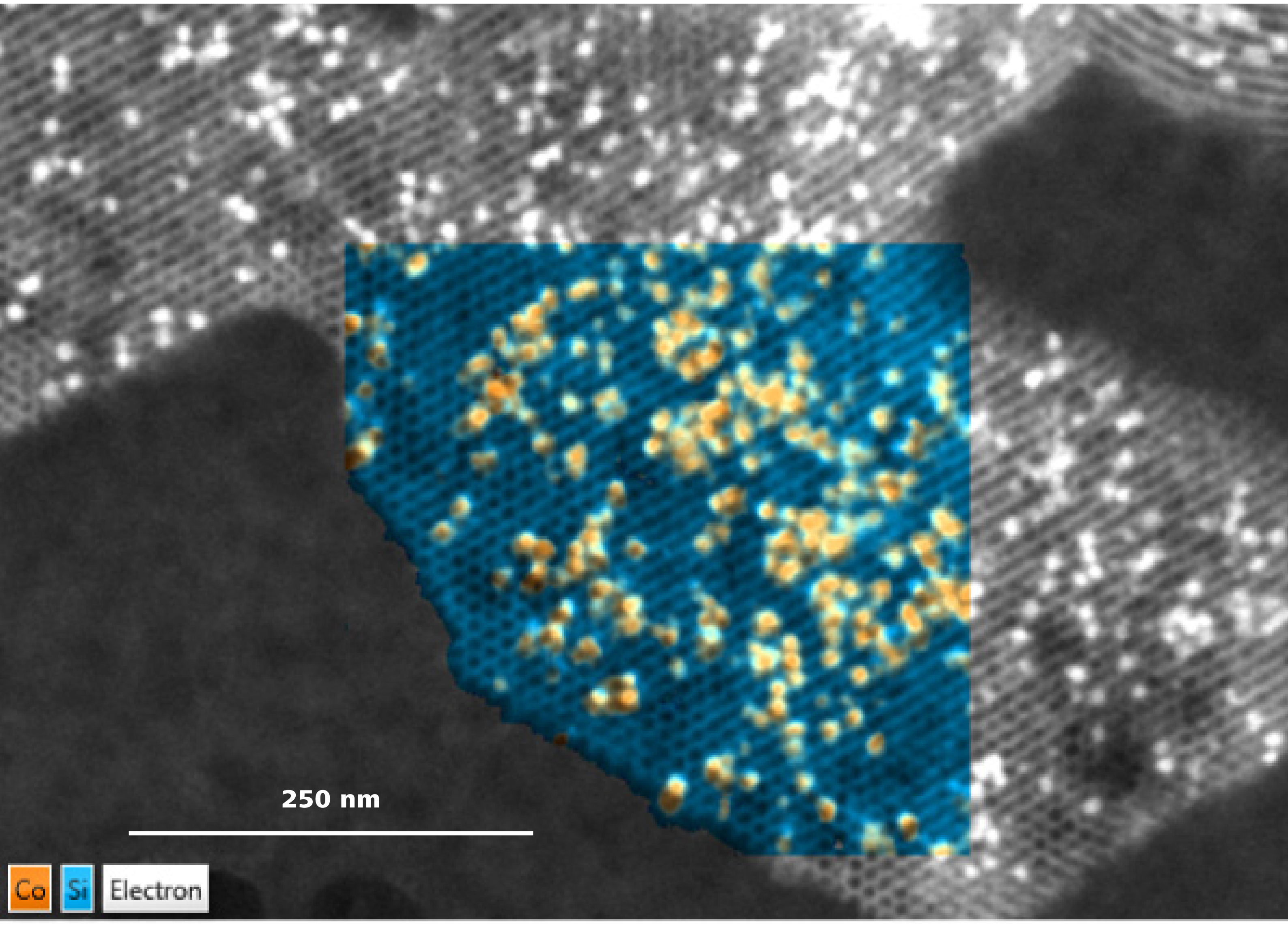

Achieving Nano-scaled EDS Analysis in an SEM

with a Detector for Transmission Scanning Electron Microscopy

Seeing beyond

Achieving Nano-scaled EDS Analysis in an SEM

with a Detector for Transmission Scanning Electron Microscopy

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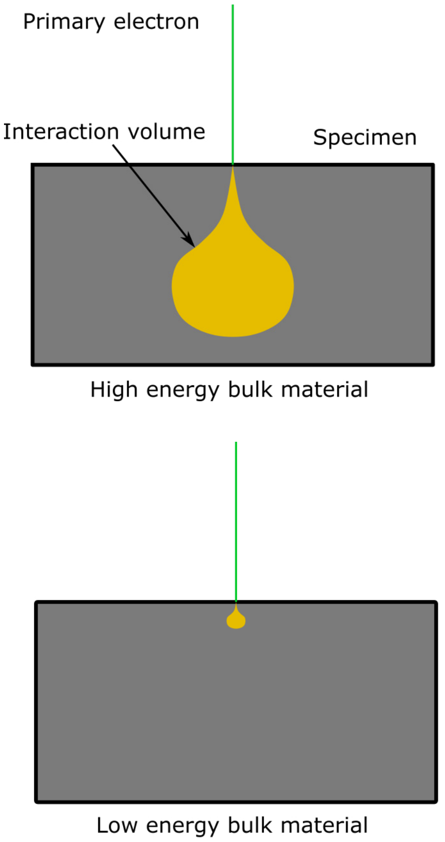
Date: October 2019

The spatial resolution of energy dispersive spectroscopy (EDS) analysis is fundamentally limited by the interaction volume of the characteristic X-ray emission. By using a thin specimen for SEM investigations, the interaction volume can be reduced in transmission scanning electron microscopy (STEM), achieving nanometer scaled resolution for EDS analysis. In this application note the details and caveats of STEM-EDS analysis will be discussed.

Introduction

The interaction volume of a characteristic X-ray emission is illustrated in figure 1. Typical EDS analysis in an SEM is performed at relatively high energy (> 10 kV), leading to a huge interaction volume in the order of micrometers.



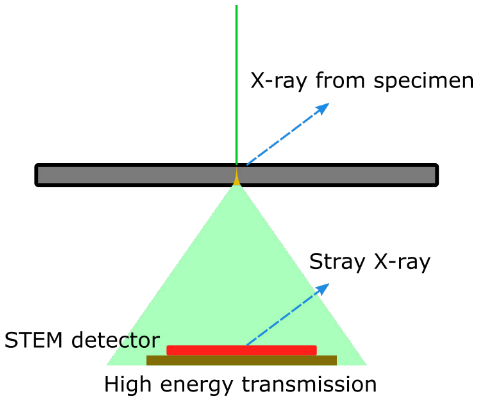


Figure 1 *Schematics comparing the interaction volume of X-ray emission in an SEM between high energy bulk, low energy bulk and high energy transmission.*

By reducing the energy of the primary electron beam, the interaction volume can be reduced significantly. With the help of a specially designed detector, EDS analysis with a beam energy down to 1 kV is feasible, demonstrating nanometer-scaled resolution [1]. However, low energy EDS faces many practical challenges: X-ray emission with low electron beam energy has a very low yield, leading to low count rates during analysis. Moreover, for higher atomic numbers low energy X-ray lines such as M and even N lines must be used. These low energy X-ray emission lines typically have complicated shapes and are very difficult to quantify. The high surface sensitivity of low energy EDS also requires a very clean specimen. An alternative method to reduce the interaction volume is to use a thin sample: in a transmission scanning electron microscope, the sample thickness is reduced to tens of nanometers in order to be transparent to electron beam when imaging. In contrast to low energy EDS, STEM-EDS benefits from the high energy of the primary electron beam to excite K or L lines, which can be easily quantified.

A practical challenge for STEM-EDS is the stray X-ray signal. In a STEM configuration the majority of the electrons penetrate through the specimen and arrives at various parts of the SEM system. This would produce pronounced stray X-ray signals, especially from Al and Si K lines, originated from the SEM stage and the STEM detectors. To reduce the stray X-ray signal a collimator should be used for the EDS detector to limit the acceptance angle of the incoming X-rays.

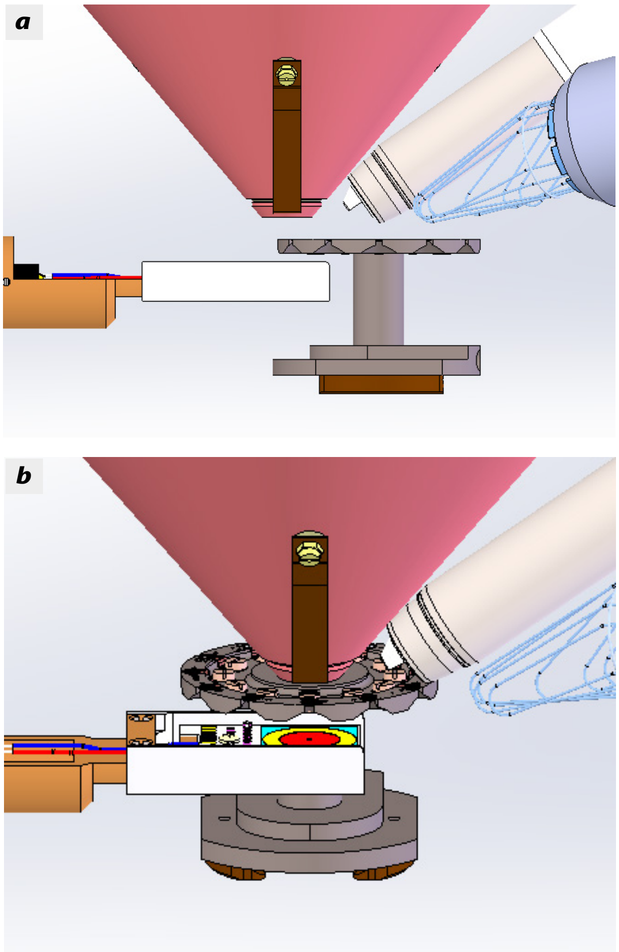


Figure 2 *Comparing a favorable (a) and unfavorable (b) configuration for an EDS analysis with STEM. Under the favorable configuration, the stray X-ray signal from the detector and sample chamber is blocked by the STEM sample holder.*

Additionally, the specimen holder for the STEM samples can be utilized to block the stray X-ray signal. This method is illustrated in Figure 2.

There are multiple possible configurations to place the STEM specimen against the STEM detector, but only one optimum to block the stray X-ray signal. In this favored configuration, the STEM specimen holder sits right below the EDS detector and above the STEM detector, and thus blocks the direct line-of-sight between the EDS detector and rest of the SEM chamber and prevents the stray X-rays from entering the EDS detector. The effectiveness of this favorable configuration can be seen in Figure 3.

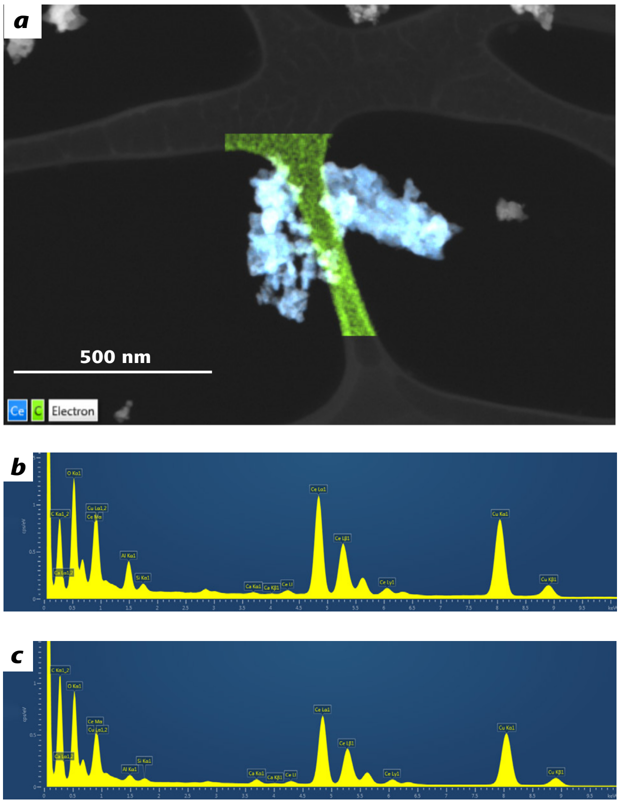


Figure 3 *High resolution EDS mapping of CeO_2 nanoparticles deposited on a carbon grid measured at 30 kV. The corresponding EDS spectrum obtained in two different configurations. In the favorable configuration (c) the stray signal from the STEM detector / sample chamber is reduced.*

The test specimen consists of CeO_2 nanoparticles deposited on the lacey carbon on a copper grid. The elemental mapping clearly demonstrates the high spatial resolution of STEM-EDS analysis. The same analysis is performed using the favorable and unfavorable configuration, respectively. Comparing the two EDS spectra shows distinctly that in the favorable configuration the stray signals from Si and Al are heavily suppressed.

The STEM-EDS technique is especially suitable for nanomaterials. Due to their nanometer-scaled size in at least one of their dimensions, they are naturally transparent to a high energy electron beam, and thus can be prepared by simply dispersing them on a typical copper grid with a carbon film. In the example shown in Figure 4, the Co nanoparticles dispersed in mesoporous silica are imaged at 30 kV in high angular dark field mode.

The Co nanoparticles have an average size around 10 nm and are deposted inside the nanometer sized channels of the mesoporous silica. The elemental mapping shown in figure 4 demonstrates the capability of STEM-EDS to resolve nanometer-scales objects even below a size of 10 nm.

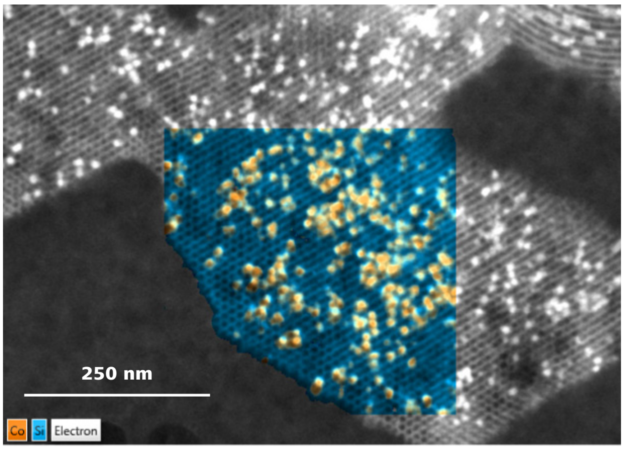


Figure 4 *High resolution EDS mapping of Co nanoparticles embedded in mesoporous silica measured at 30 kV. Individual Co nanoparticles with approx. 10 nm size are resolved.*

References:

[1] Nanometer scale EDS Analysis using Low-kV FE-SEM and Windowless EDS Detector, Technology Note ZEISS.

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